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14. ABSTRACT  This report results from a contract tasking E.O.Paton Electric Welding Institute as follows: Because TiAl-base materials have relatively low density, high strength, good wear resistance, and excellent resistance to oxidation and corrosion, they are of considerable interest in the aerospace industry. In particular, the materials are prime candidates for components aircraft engines. However, the relatively low plasticity of the intermetallic compound presents challenges with respect to the fabrication of small/thin cross-section semi-finished products such as rod, sheet, foil and so on. Hence, many of the applications of the materials are limited at present. When TiAl products of are made by conventional thermomechanical processing of ingots, the formation of the mechanical and/or crystallographic texture, the segregation of impurities to grain boundaries by impurities, etc., may adversely affect the performance of the finished product.					
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# E.O.Paton Electric Welding Institute

## FINAL REPORT (Project P-339)

“Determine the conditions of formation of  
defect-free TiAl foil”

Project Manager

Prof. A. I. Ustinov

Kiev 2008

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## **Summary**

At the final stage of the work we focused on the possibility of producing defectfree foils with the surface size of  $150 \times 200 \text{ mm}^2$  based on TiAl alloy with Cr and Nb additives. During work performance in the second stage it was established that because of the difference in the distance to the axis of revolution, the thickness of different sections of the foil differs significantly. Therefore, in order to produce foil of a more uniform thickness, an impermeable screen was installed in the chamber, ensuring shadowing of foil sections located closer to the axis of revolution of the substrate holder. Conducted research showed that such geometry of the vapour flow deposition provides a lowering of foil non-uniformity across the thickness. It is shown that element distribution across the foil thickness does not depend on the location of the section on its surface. In order to obtain Ti-Al-Cr-Nb condensates we used three evaporators, accommodating ingots of Ti-Cr, Al and Nb. With preservation of stable operation of the unit using this schematic (three evaporators and a substrate rotating above them about a vertical axis), we produced condensates with a uniform distribution of elements across the thickness. Investigation of condensate microstructure in the initial condition showed the presence of a laminated substructure on the nanoscale. The foils were sent to the Partner.

Summarizing the obtained in this work results we can conclude that the relatively thin intermetallic foils on the basis Ti-Al and Ti-Al-Cr-Nb systems with the specified composition and thickness can be produced by electron beam evaporation of components from the ingots placed into the water-cooled crucibles and co-deposition of vapor flows on rotated substrate.

## **Methods, Assumptions, and Procedures**

1. Producing a condensate of Ti-Al system intermetallic with an improved uniformity of thickness.

Condensate deposition on a rotating substrate creates a non-uniformity of thickness related to the fact that in the point of maximum removal of the substrate from the evaporation source the distance from the source to the nearest substrate edge (point A Fig. 1) is smaller than the distance to the farther edge (point B Fig. 1). As the vapour flow intensity decreases in proportion to the square of the distance, the thickness of the deposited condensate in point A turns out to be greater than condensate thickness in point B. Partial shadowing of the vapour flow (complete shadowing of the flow to point A and complete transmission to point B) using a screening partition was used to reduce this non-uniformity.

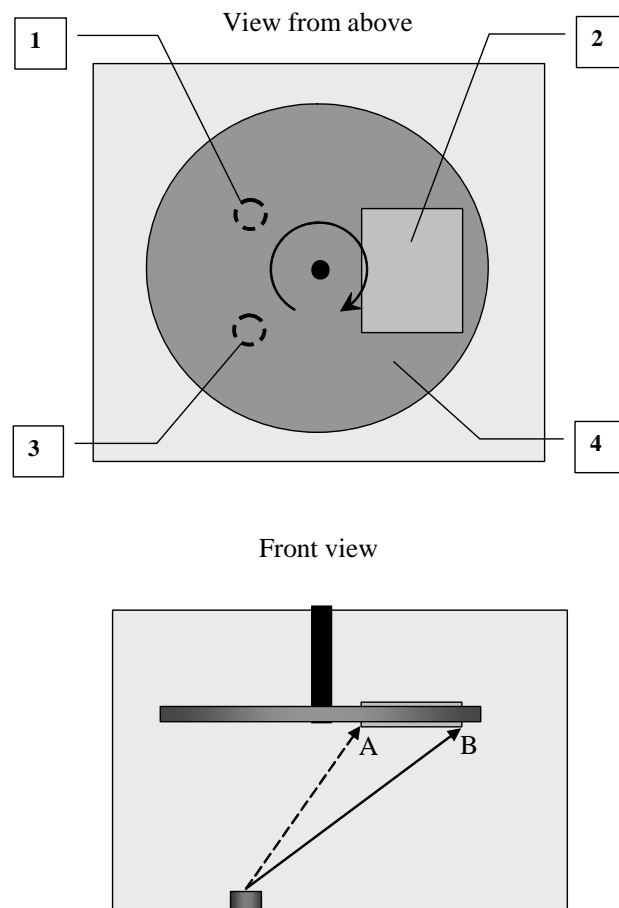


Fig.1. Schematic of electron beam evaporation and scheme of substrates holder (conventional design): 1-ingot Al, 2-substrate, 3-ingot Ti, 4-rotating holder

Schematic of the vacuum chamber to produce Ti-Al intermetallic with an improved uniformity is given in Fig. 2. Condensate of Ti-Al intermetallic was deposited by the method of electron beam evaporation from two ingots of Ti and Al (1 and 3). The substrates (2) used were high-temperature steel plates (150x200 mm size) fastened on a rotating holder (4). Substrate rotation rate during the process was constant and equal to 30 rpms. Substrate heating was performed using two auxiliary electron beam evaporators (not shown in the schematic). Substrate temperature during the condensate deposition was equal 850-900°C.

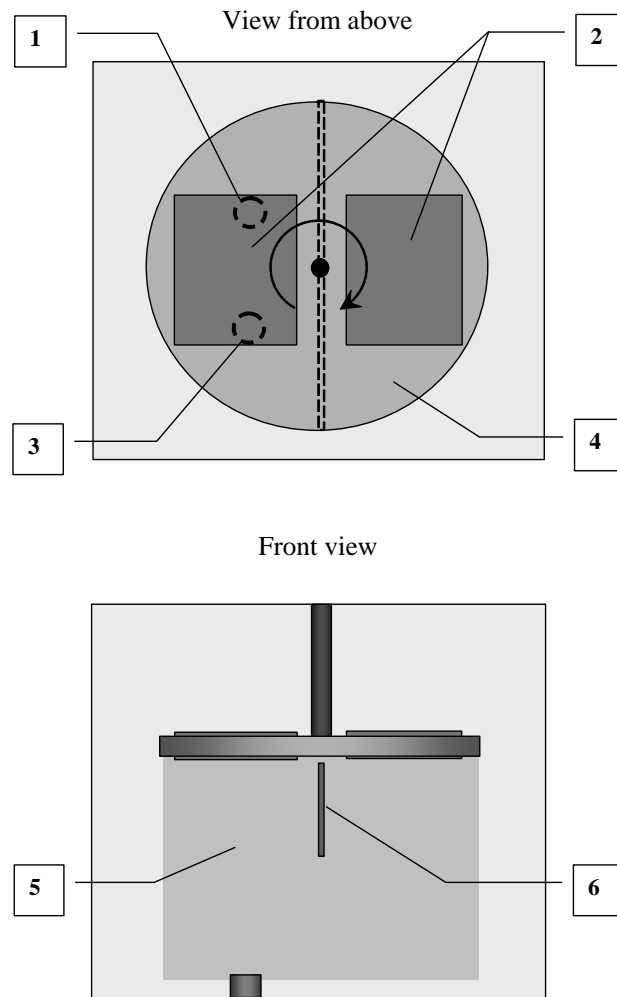


Fig 2. Schematic of electron beam evaporation and scheme of substrates holder with dividing partition: 1 - ingot Al, 2 – substrate, 3 - ingot Ti, 4 - rotating holder, 5- vapour flow, 6 - dividing partition

Ti and Al ingots were placed into water-cooled crucibles located under the substrate at 0.3 m distance. During evaporation the ingots were lifted at a constant rate, which was selected to be such for each ingot that the distance from the pool surface to the substrate was constant.

A layer of  $\text{CaF}_2$  (2-4 mcm) salt was first deposited on the substrate to simplify condensate separation. At condensate cooling, because of the great difference in the coefficients of thermal expansion (CTE) of the salt and condensate, the condensate separated along the salt-condensate boundary.

A screening partition (6) was used for partial shadowing of the vapour flow (5).

## 2. Producing Ti-Al intermetallic condensate with Nb and Cr additives

Schematic of the vacuum chamber for producing Ti-Al intermetallic with Nb and Cr additives is shown in Fig. 3.

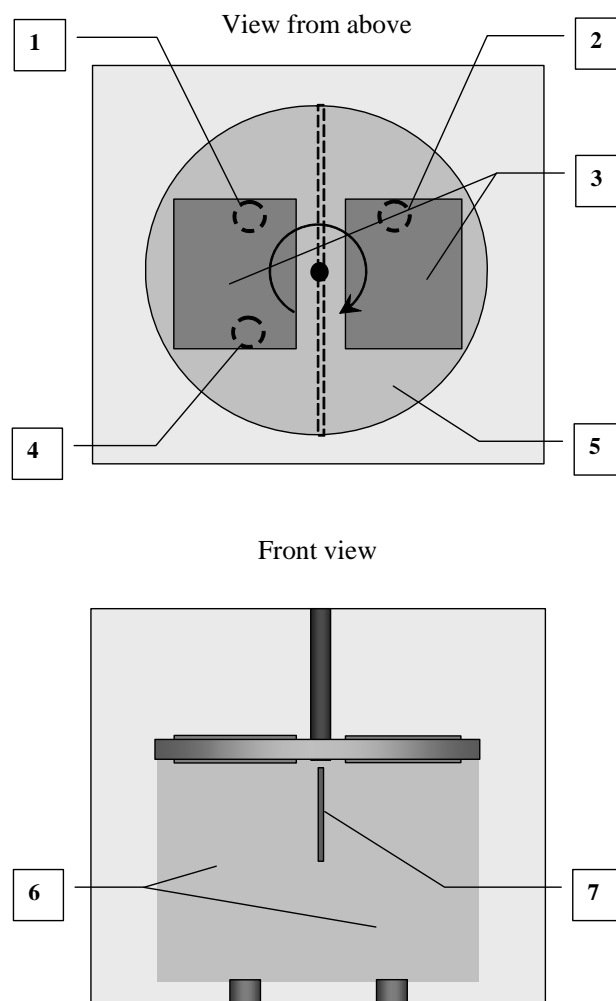


Fig 3. Schematic of electron beam evaporation and scheme of substrates holder with dividing partition: 1 - ingot Al, 2 - ingot Nb, 3 – substrate, 4 - ingot Ti-Cr, 5 - rotating holder, 6 - vapour flow, 7 - dividing partition



The condensate was deposited by the method of electron beam evaporation of three ingots Ti-Cr (4), Al (1) and Nb (2). The substrates used were two plates of high-temperature resistant steel (150x200 mm size) fastened on a rotating holder (5). The rate of substrate rotation during the process was constant at 30 rpm. Substrate heating was performed using two auxiliary electron beam evaporators (not shown in the schematic). Substrate temperature during condensate deposition was equal to 850 – 900°C.

Ti and Al ingots were in water-cooled crucibles located under the substrate at 0.3 m distance. During evaporation the ingots were raised at a constant rate, which was selected for each ingot to be such that the distance from the pool surface to the substrate was constant.

To simplify condensate separation a layer of CaF<sub>2</sub> salt (2-4 mcm) was first applied on the substrate. At condensate cooling condensate separation occurred along the salt-condensate boundary due to the great differences in the coefficients of thermal expansion (CTE) of the salt and the condensate.

## Results and Discussion

Application of the above screening partition allowed a certain leveling of condensate thickness over the area. Fig. 4 shows examples of condensate thickness distribution over its area under the conditions of condensation without (1) and the screening partition (2).

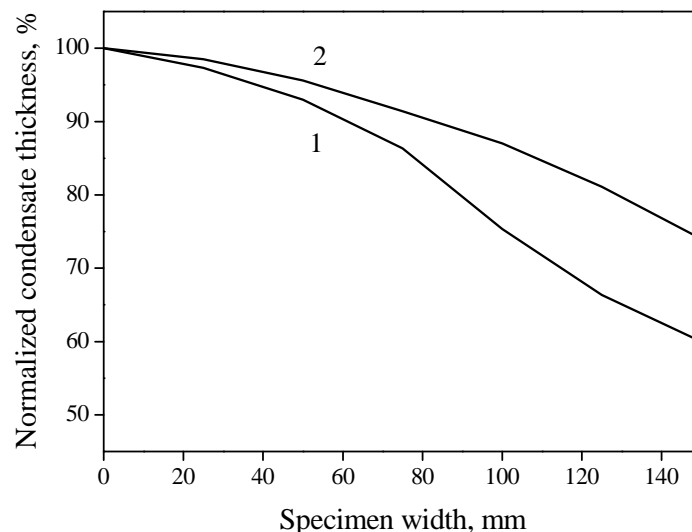


Fig. 4. Distribution of condensate thickness over the area. 1- in conditions of condensation without screen; 2- in conditions of condensation with screening partition

Thus, application of a screening partition allows adjustment of the vapour flow at its condensation on the substrate, thus providing condensate leveling by thickness. Condensates of equal thickness can be produced by modeling the shutter geometry.

The main goal during fulfillment of the third stage was producing a condensate of TiAl intermetallic alloyed by Cr and Nb with uniform distribution of alloying elements across the thickness. This goal was successfully achieved using three evaporators: Ti-Cr, Al and Nb following the procedure described above. A characteristic distribution of elements across the condensate thickness is shown in Fig. 5.

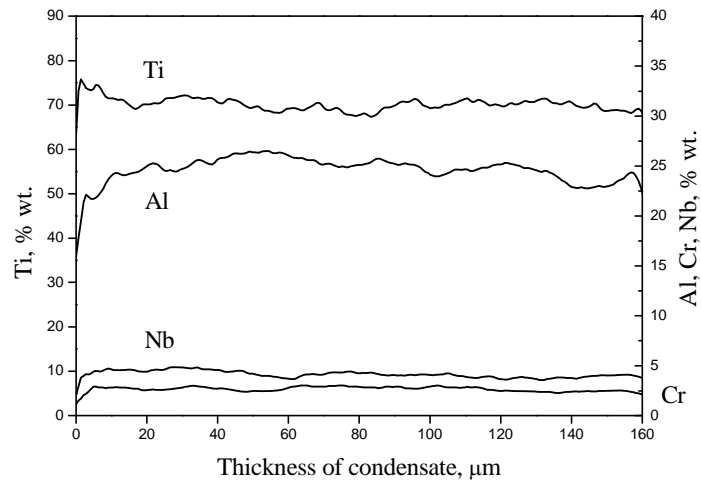


Fig. 5. Distribution of component concentration across the condensate thickness.

It is seen that the proposed condensation schematic provides a uniform distribution of components across the condensate thickness and the required composition. This resulted in producing condensates, the composition of which corresponds to the two-phase region (TiAl+Ti<sub>3</sub>Al) with different Ti/Al ratios. Condensate microstructures are given in Fig. 6.

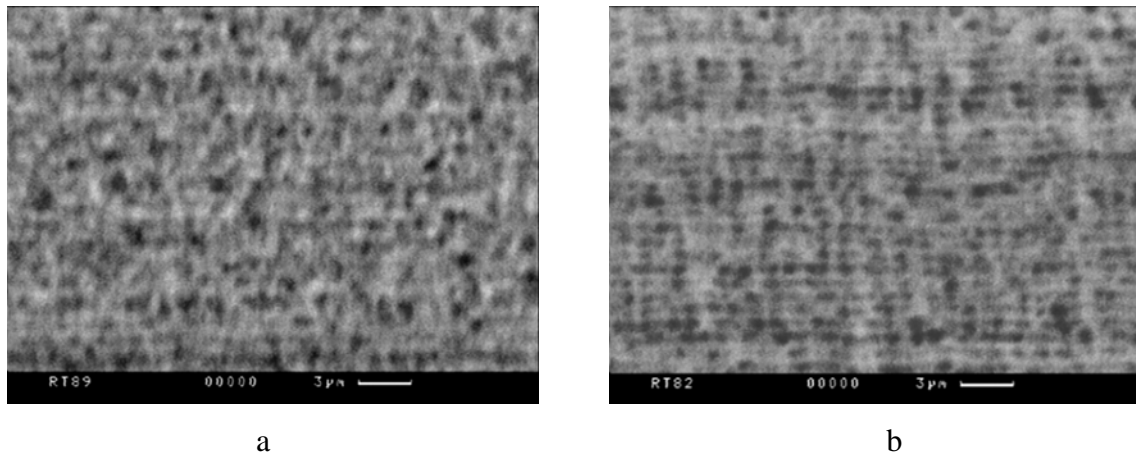
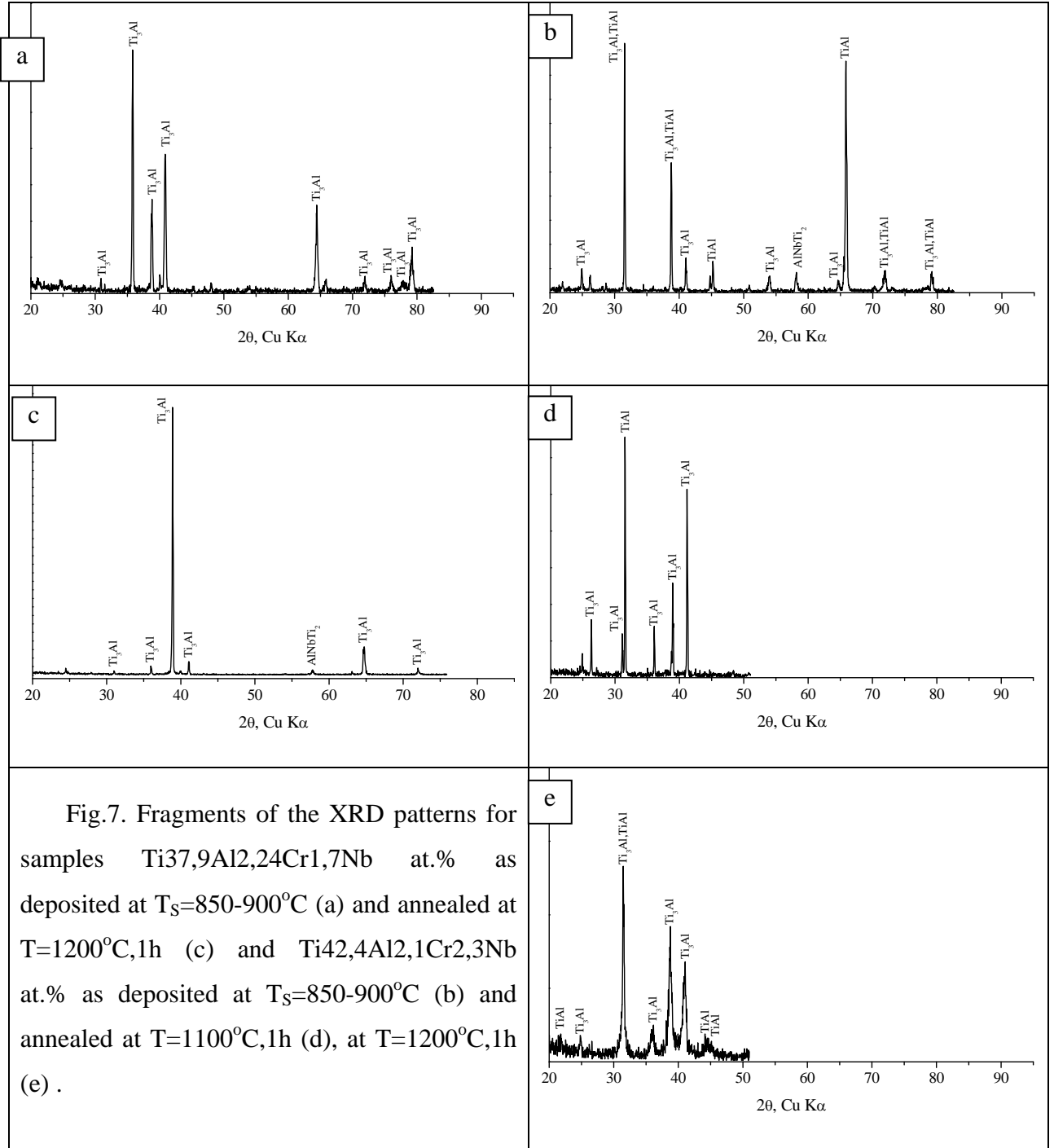


Fig.6. Microstructure of Ti<sub>37,9</sub>Al<sub>2,24</sub>Cr<sub>1,7</sub>Nb at.% (a) and Ti<sub>42,4</sub>Al<sub>2,1</sub>Cr<sub>2,3</sub>Nb at.% (b) condensates deposited at the temperatures of 850-900°C

As shown by X-Ray phase analysis the produced condensates consist of a mixture of  $Ti_3Al$  and  $TiAl$ , the volume fractions of which depend on the condensate composition – with increase of aluminium content the volume fraction of  $TiAl$  phase rises. The respective diffractograms are given in Fig. 7(a,b).



It is seen that the condensates in the initial condition are characterized by residual lamination related to instability of the vapour flows at substrate displacement above the evaporators with different ingots. As was shown earlier, annealing of condensates at the temperature of 1000° C, 2 h, allows an essential reduction of their lamination, without, however, eliminating it completely. Annealing directly in the chamber requires essential technological optimization, and its duration is limited.

To optimize the regime of annealing treatment, ensuring elimination of residual lamination and formation of structurally homogeneous condensates, annealing of condensates with different composition at 1100 и 1200 °C, 1 hour was performed. To prevent condensate distortion at cooling, they were placed between molybdenum plates. It should be noted that no molybdenum was found on the annealed condensate surface. Fig. 8 gives the microstructures of annealed condensates of different composition. One can see that annealing at 1100°C leads to elimination of lamination. The annealing temperature substantially affect on condensates microstructure. So, annealing at 1100°C leads to two-phase structure (TiAl+Ti<sub>3</sub>Al) formation (see fig.7 ) with high dispersion of phases (fig.8c,d). Annealing at 1200°C leads to roughening of Ti<sub>3</sub>Al particles and lamel structure formation.

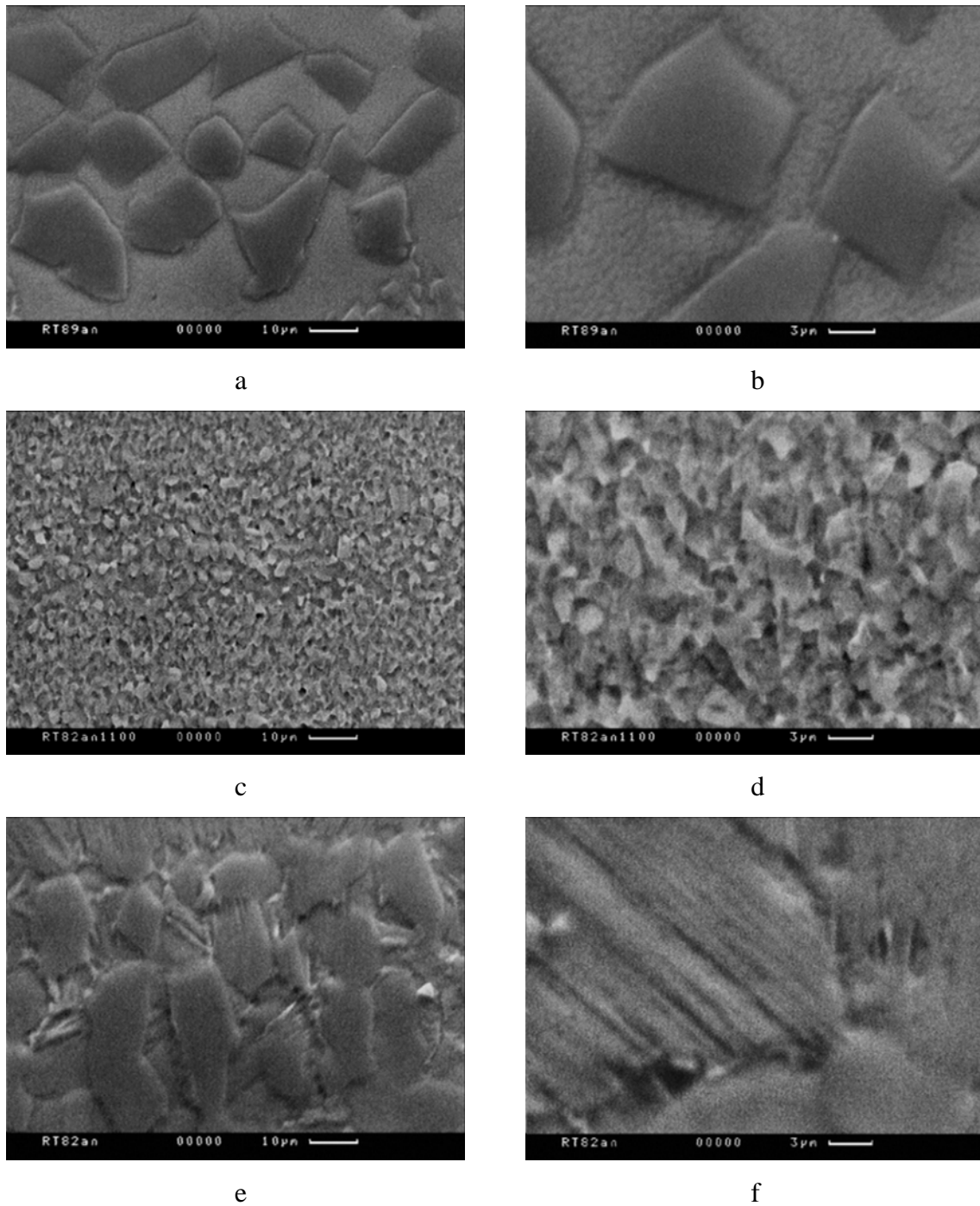


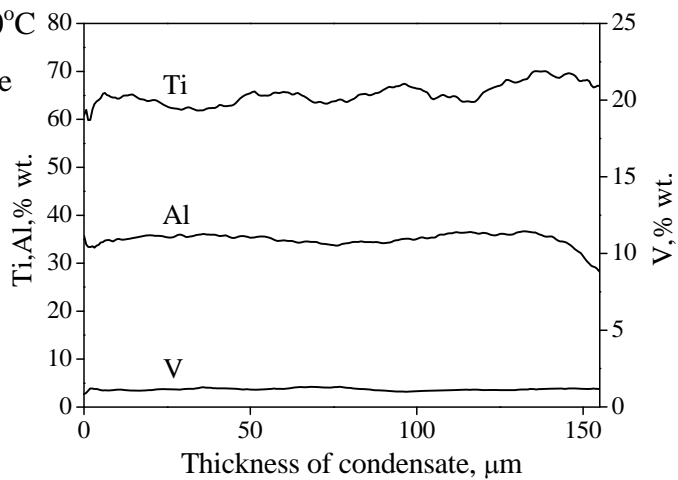
Fig. 8. Microstructures of Ti<sub>37,8</sub>Al<sub>2,24</sub>Cr<sub>1,7</sub>Nb condensate, annealed at 1200°C, 1 h (a,b) and Ti<sub>42,4</sub>Al<sub>2,1</sub>Cr<sub>2,3</sub>Nb condensate, annealed at 1100°C, 1 h. (c,d), at 1200°C, 1 h (e,f)

It is seen that the structure of annealed condensates depends on their composition. In condensates of a composition close to the lower boundary of the two-phase region (TiAl+Ti<sub>3</sub>Al) coarse Ti<sub>3</sub>Al particles form during annealing (Fig. 8 a, b). Annealing of aluminium enriched condensates, alongside formation of coarse Ti<sub>3</sub>Al particles, leads to formation of a lamellar structure (Fig. 8 e, f), which is known to lead to improvement of the condensate mechanical properties.

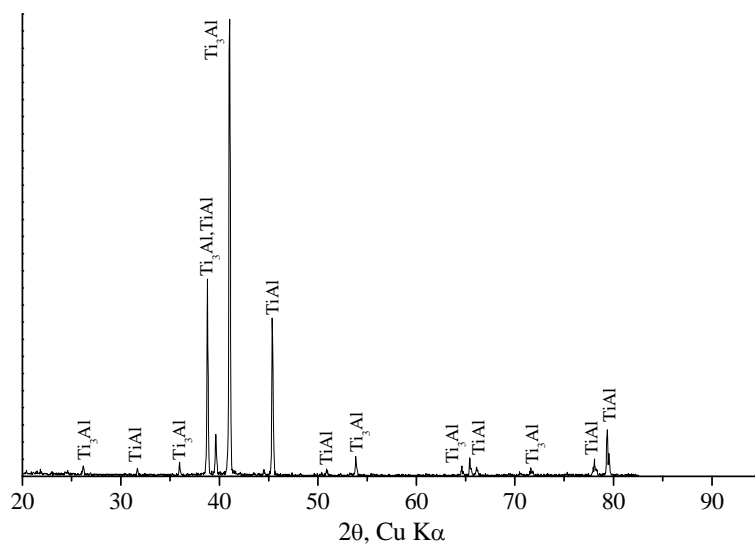
## Characteristics of condensates with a large surface area.

### Condensate #1(RT71)

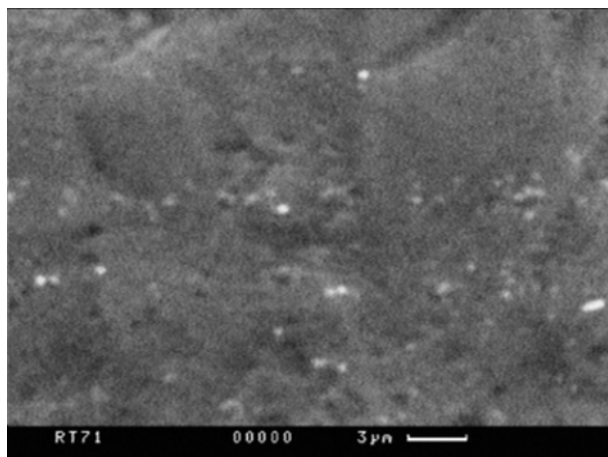
Thickness of condensate 155  $\mu\text{m}$ ,  $T_s = 850^\circ\text{C}$   
Elements distribution across the condensate  
thickness



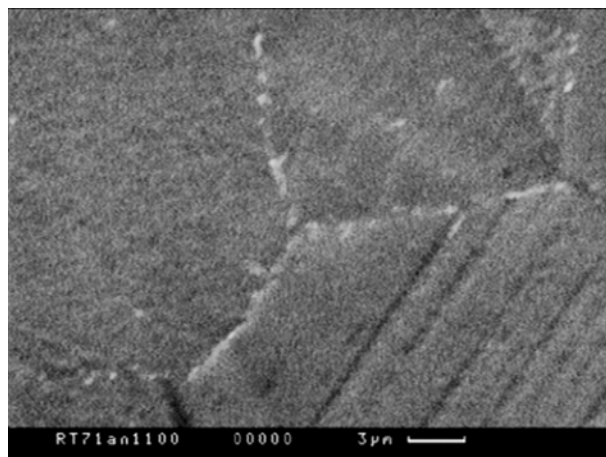
Fragment of the XRD pattern for  
condensate as annealed at  
 $1100^\circ\text{C}$ , 1h



### Microstructure of condensate



As deposited

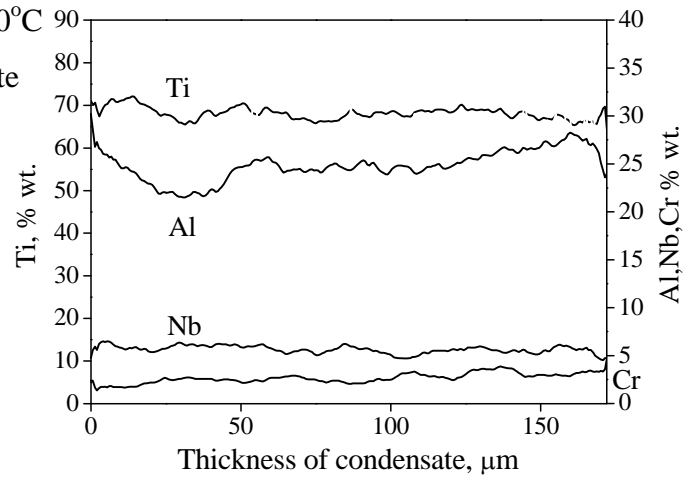


As annealed at  $1100^\circ\text{C}$ , 1 h

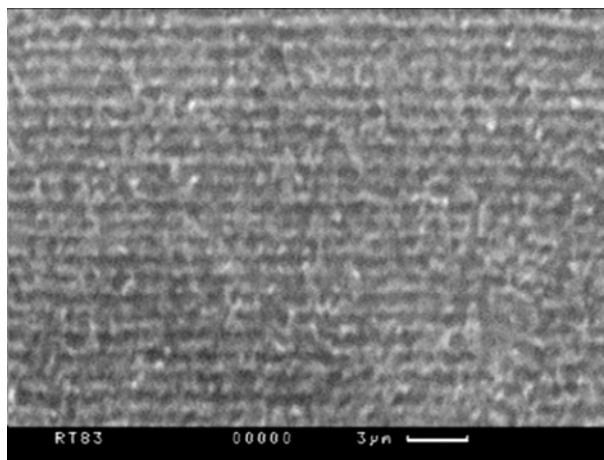
## Condensate #2(RT83)

Thickness of condensate 170  $\mu\text{m}$ ,  $T_s = 850^\circ\text{C}$

Elements distribution across the condensate thickness

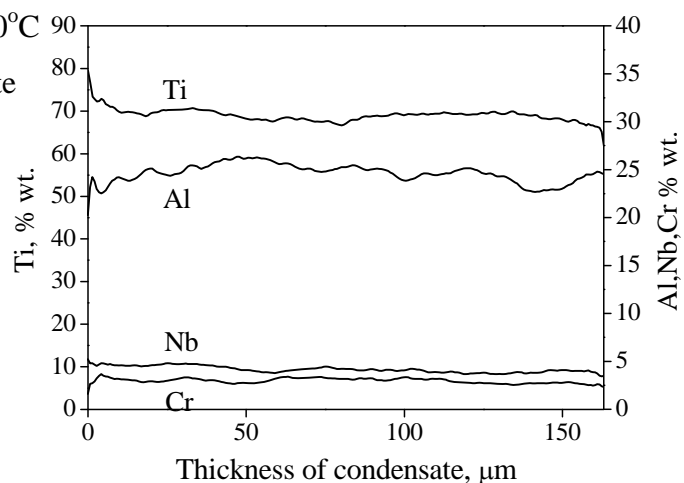


Microstructure of condensate as deposited

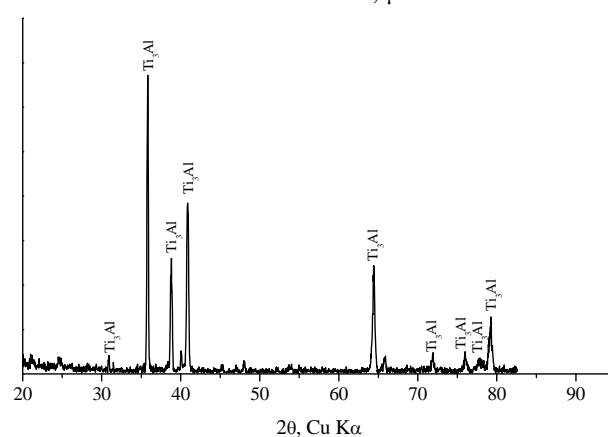


### Condensate #3(RT89)

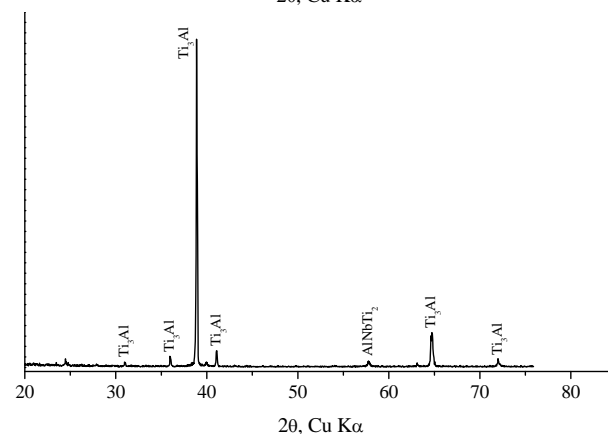
Thickness of condensate 160  $\mu\text{m}$ ,  $T_s = 850^\circ\text{C}$   
Elements distribution across the condensate  
thickness



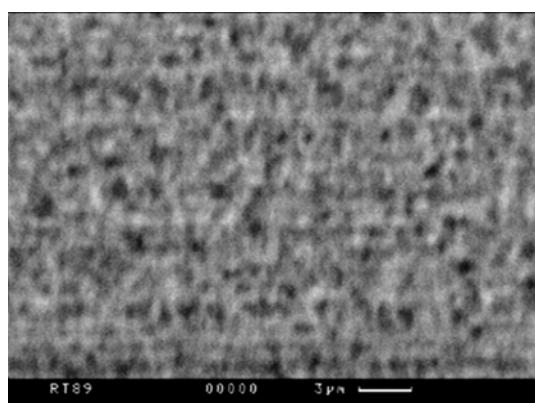
Fragment of the XRD pattern for  
condensate as deposited



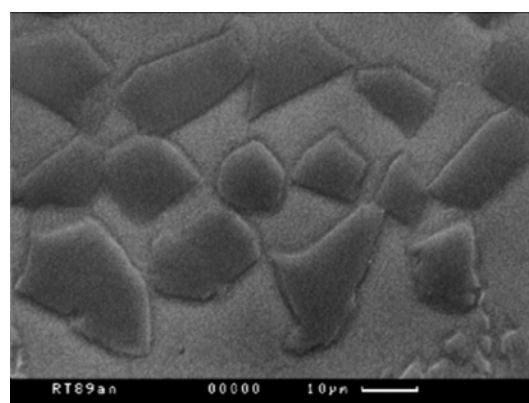
Fragment of the XRD pattern for  
condensate as annealed at  
1200°C, 1h



Microstructure of condensate



As deposited



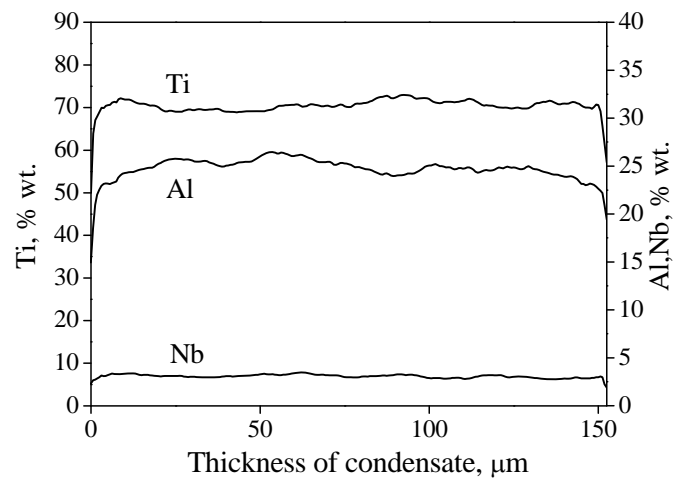
As annealed at 1200°C, 1h



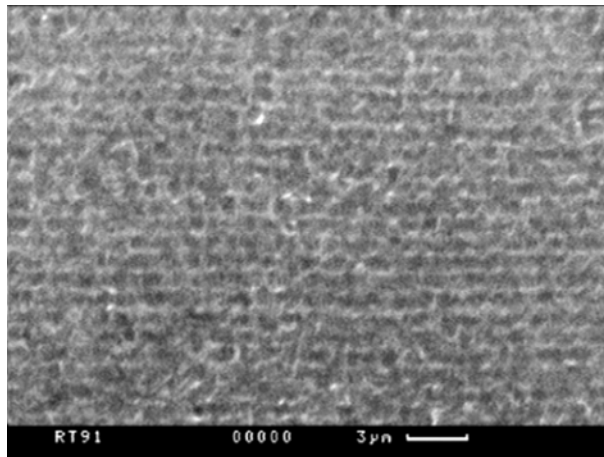
## Condensate #4(RT91)

Thickness of condensate 152  $\mu\text{m}$ ,  $T_s = 850^\circ\text{C}$

Elements distribution across the condensate thickness

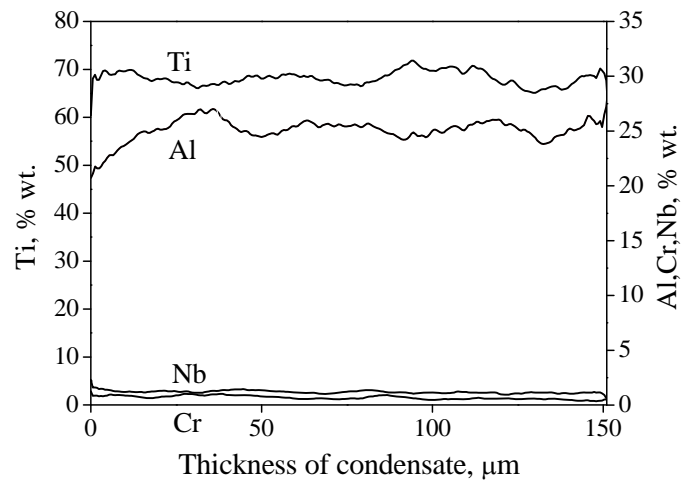


Microstructure of condensate as deposited

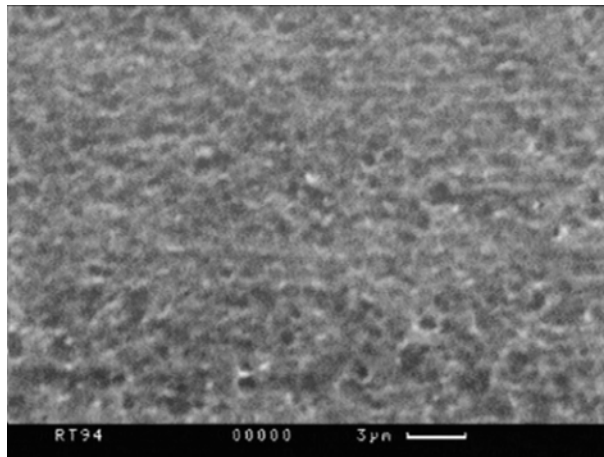


### Condensate #5(RT94)

Thickness of condensate 150  $\mu\text{m}$ ,  $T_s = 850^\circ\text{C}$ . Elements distribution across the condensate thickness



Microstructure of condensate as deposited



## Conclusions

1. The using of additional screen in vacuum chamber, which shadows part of the substrate, allows to improve the uniform of thickness of Ti-Al foil which is forming during EBPVD process.
2. The intermetallic foils 150 x 200 mm<sup>2</sup> surface and thickness of up to 200 micron on the basis of Ti-Al-Cr-Nb system of the specified composition with uniform distribution of component across the entire their thickness were obtained by co-deposition on rotated substrate vapor flows produced by electron beam evaporation of Ti-Cr, Al and Nb ingot.
3. Thermal treatment of foils obtained by EBPVD method allow to improve uniform component distribution across of the entire their thickness and affect on their microstructure characteristics.
4. Obtained in this work results demonstrate that the relatively thick intermetallic foils on the basis Ti-Al and Ti-Al-Cr-Nb systems with the specified composition and thickness can be produced by the method of electron beam evaporation of components from the ingots placed into the water-cooled crucibles and condensation of vapor flows of their component on rotated substrate.

## **List of Symbols, Abbreviations, and Acronyms**

EBPBD – electron-beam physical vapor deposition

$T_s$  - condensation temperature

XRD - X-ray diffraction